

FURACA #03/97 (~~4-15~~)

Aim: Preparation of Furaca from  
New route.

eq. to 100 gm. 2ACA.

## I Preparation of TFA

### RAW MATERIALS

2-Furyl chloride	: 46.0ml
DMW	: (700 + 30)ml
NaSH (25% excess)	: 75.0gm
EtOAc	: 500.0ml
1:1 HCl	: 95.0ml
DMW	: 350.0ml
NaHCO <sub>3</sub>	: 39.0gm
EtOAc	: 200.0ml
1:1 HCl	: 82.0ml

### Procedure

1. ~~charged~~ DMW, NaSH were charged at RT  
- Stir to get clear solution (flush i 30ml DMW)
2. Add 2-furyl chloride in 40-41' at 20-25°C
2. Stirred for 5', monitor the reaction
4. Charged EtOAc, then brought pH to 1.0-0.9  
by 1:1 HCl at 20-25°C in 10-15'
5. Separated the layers. Gave sample & oil
6. To that oil added DMW, Adjusted to 7.0-7.5  
pH by NaHCO<sub>3</sub> at 20-25°C
- Separated the layers
8. Added EtOAc to the ~~org~~ aqueous phase  
again brought pH to 1.0-0.9 by 1:1 HCl  
at 20-25°C
- = Separated the layers. Sampled for each  
layers.
10. Org. phase ready for next step.

## II preparation of Furan:

### Raw materials

7ACA	:	103.0 gm.	
EtOAc	:	400.0 ml.	
HOAc	:	60.0 ml.	
BF <sub>3</sub>	:	144.0 gm.	7 gm. extra Br Here input adjusted
TEA	:	260.0 ml.	
Hydrolysis { DMUO	:	300.0 ml.	
SHS	:	2.0 gm.	
20% NH <sub>3</sub> soln	:	184.0 ml.	
DMUO	:	(100 + 300 + 100) ml	} washing * (Slurry-wash)
EtOAc	:	(100 + 300 + 100) ml	

### Procedure

1. charge 7ACA into the system having BF<sub>3</sub> purged in EtOAc + HOAc mixture at 20°C
2. Add TEA solution as prepared before
3. maintain the temp at 30°C for 2 hrs.

7ACA	Furan	Imp	TEA
25	3.	82.57%	2.

the 35

4. After completion of reaction, transfer the mass into the DMUO (precooled at 15°C) with SHS
5. Adjust the pH to 3.5 by 20% NH<sub>3</sub> at 20-25°C in 15-25-30'
6. Stir for 3d at 20-25°C
7. Filter & wash with DMUO & EtOAc

NOTE:

Instead of 137.0 gm of  $\text{BF}_3$ , purged  
144.0 gm of  $\text{BF}_3$ . So that added 10 gm  
FACA.